

183G03-File 1

ANNEX 5

Stability Report # 183G03  
SU010308 capsic

**ACCO. USA**  
WHEELING, ILLINOIS 60090

15044



0505 15044 1  
EN VERT FEUILLE/VERDE SUAVE

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 116305  
Sample ID (Request No.): 20021134

User sample ID (lot): 183G03

Sample Type: SU010358 (PNU290940AD) CAPSULE  
25MG

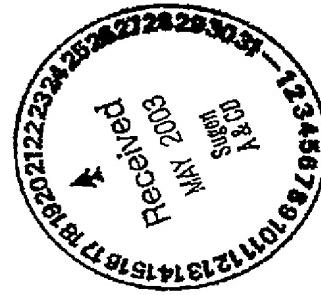
Samples Notes:

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCO02#00	CAPSULE APPEARANCE	0=UNCHANGED	1	bx				15545/18- MAY 2002	CAPPUCIO	MARIANI
	CAPSULE COLOR	0=UNCHANGED	1	bx				15545/18- MAY 2002	CAPPUCIO	MARIANI
	CONTENT APPEARANCE	0=UNCHANGED	1	bx				15545/18- MAY 2002	CAPPUCIO	MARIANI
	CONTENT COLOR	0=UNCHANGED	1	bx				15545/18- MAY 2002	CAPPUCIO	MARIANI
	SMELL	0=UNCHANGED	1	bx				15545/18- MAY 2002	CAPPUCIO	MARIANI
083GST01#01_S	(HPLC TITER) SAMPLE#1	97.95	1	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
	(HPLC TITER) SAMPLE#2	97.96	1	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
	(HPLC TITER) SAMPLE#3	97.25	1	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
	AVERAGE TITER (HPLC)	97.72	1	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
083GSC01#00	TOTAL CORRELATED SUBSTANCES	0.18	1	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	1	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
083GKF01#00	(HUMIDITY) SAMPLE #1	2.37	1	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	(HUMIDITY) SAMPLE #2	2.4	1	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	(HUMIDITY) SAMPLE #3	2.38	1	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	AVERAGE HUMIDITY	2.38	1	%				15545/18- MAY 2002	CAPPUCIO	MARIANI



# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 116305  
Sample ID (Request No.): 20021134

User sample ID (lot): **183G03**

Sample Type: SU010398 (PNU290940AD) - CAPSULE  
25MG

### Samples Notes:

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#01	SAMPLE#1	94.01	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SAMPLE#2	99.04	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SAMPLE#3	100.16	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SAMPLE#4	94.01	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SAMPLE#5	88.41	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SAMPLE#6	94.56	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	DISSOLUTION	95.03	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	STANDARD DEVIATION	4.208	1	std-1				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	C.V.	4.43	1	%				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	MIN. DISSOLUTION VALUE	88.41	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	MAX. DISSOLUTION VALUE	100.16	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI

Status :

**COMPLETE**

# PHARMACIA

Global Pharmaceutical Sciences - Italy



## Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 116305  
Sample ID (Request No.) : 20021134

User Sample ID (lot) : IR3G03  
Sample Type: SU010398 (PNU280840AD) CAPSULE  
25MG

Request Type: STABILITY  
Stability Study: IR3G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

### Samples Notes:

#### Observations on the Method

083GC02#00, component: CAPSULE APPEARANCE

Opaque brick red colored, format:3, hard gelatin capsule containing yellow-orange powder

#### Observations on the Method

083GDS01#01, component: DISSOLUTION

Dissolution at 15 minutes: 76.5% (C.V.=13.9%)

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116305  
Sample ID (Request No.): 20021134  
User sample ID (lot): 183G03  
Sample Type: SU010398 (PNU280840AD) CAPSULE  
25MG

Samples Notes:

Observations on the Method 083GDS01#01, component: DISSOLUTION  
Dissolution at 45 minutes: 99.97% (C.V.=1.16%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS than 0.05%

Total correlated substances: 0.18%

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116805  
Sample ID (Request No.): 20021134  
User sample ID (lot): 183G03  
Sample Type: SU01039 (PNU290940AD) CAPSULE  
25MG

Request Type: STABILITY

Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:

APPROVED

Status:

COMPLETE

### Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116305

Sample ID (Request No.): 20021134

User sample ID (lot): **I83G03**

Sample Type: SUB10398 (PNU29094DAD) CAPSULE  
25MG

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

RRT Area (%)

Request Type: STABILITY  
Stability Study: I83G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:

APPROVED

Status:

COMPLETE

# PHARMACIA



Global Pharmaceutical Sciences - Italy

## Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 116305  
Sample ID (Request No.): 20021134  
User sample ID (lot): **I83G03**  
**Sample Type:** SU010998 (PNU230940AD) CAPSULE  
25MG

### Samples Notes:

Observations on the Method: 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Request Type: STABILITY  
Stability Study: I83G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116805

Sample ID (Request No.): 20021134

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU280940AD) CAPSULE

25MG

Request Type: STABILITY

Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/50% U.R.

Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.66 0.12

sytollandt

Printed 20-MAY-2003 01:53 PM by GIUSEPPE MACCARI

Page 8 / 10

# PHARMACIA

Global Pharmaceutical Sciences - Italy



## Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 116305  
Sample ID (Request No.): 20021134

User sample ID (lot): 1B3G03

Sample Type: SU010398 (PNU280940AD) CAPSULE  
25MG.

Request Type: STABILITY  
Stability Study: 1B3G03

Pick Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
APPROVED

Status:  
COMPLETE

### Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.91    0.08

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 116305  
Sample ID (Request No.): 20021134

User Sample ID (lot): 183G03  
Sample Type: SU010398 (PNU290840AD) CAPSULE  
25MG

Request Type: STABILITY  
Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.

Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition :  
**APPROVED**

Status :  
**COMPLETE**

### Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

1.00 99.82 (SU1124B)

SUBMISSION APPROVED BY Volker ECK  
APPROVE DATE 15-may-2003

SAMPLE APPROVED BY: ROSARIA MARIANI  
APPROVE DATE: 17-may-2002

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

### Global Pharmaceutical Sciences - Italy

Submission Id: 116307

Sample ID (Request No.): 200211138

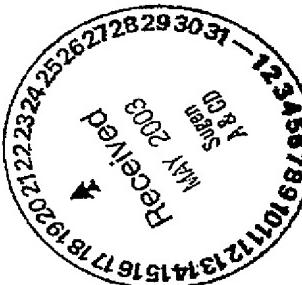
User sample ID (lot): 083G03

Sample Type: SU010388 (PNU280840AD) CAPSULE  
25MG

#### Samples Notes:

**Request Type: STABILITY**  
 Stability Study: 083G03  
 Pack Type: 30 ml PLASTIC BOTTLE  
 Storage Condition: 40°C/75% U.R.  
 Time Label: 6 MONTHS  
 Preparation purpose: CLINICAL SUPPLY + STABILITY

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCC02#0	CAPSULE APPEARANCE	0=UNCHANGED	1	txt				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	CAPSULE COLOR	0=UNCHANGED	1	txt				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	CONTENT APPEARANCE	0=UNCHANGED	1	txt				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	CONTENT COLOR	0=UNCHANGED	1	txt				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SMELL	0=UNCHANGED	1	txt				15545/18- MAY 2002	CAPPUCCIO	MARIANI
083GTT01#01_S	(HPLC TITER) SAMPLE#1	97.43	1	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCCIO	MARIANI
	(HPLC TITER) SAMPLE#2	98.55	1	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCCIO	MARIANI
	(HPLC TITER) SAMPLE#3	97.23	1	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCCIO	MARIANI
	AVERAGE TITER (HPLC)	97.74	1	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCCIO	MARIANI
083GSC01#00	TOTAL CORRELATED SUBSTANCES	0.21	1	%				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	1	%				15545/18- MAY 2002	CAPPUCCIO	MARIANI
083GKF01#00	(HUMIDITY) SAMPLE #1	2.77	1	%				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	(HUMIDITY) SAMPLE #2	2.9	1	%				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	(HUMIDITY) SAMPLE #3	2.8	1	%				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	AVERAGE HUMIDITY	2.82	1	%				15545/18- MAY 2002	CAPPUCCIO	MARIANI



# PHARMACIA



Global Pharmaceutical Sciences - Italy

## Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 116307  
Sample ID (Request No.) : 20021136  
User sample ID (lot) : **183G03**  
**Sample Type:** SU010398 (PNU280940AD) CAPSULE  
25MG

Samples Notes:

Request Type: STABILITY  
Stability Study: IBSG03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 40°C/75% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Samples Notes:

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analysis	Analyst	Venier
083GDS01#01	SAMPLE#1	96.29	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SAMPLE#2	95.75	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SAMPLE#3	96.82	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SAMPLE#4	100.05	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SAMPLE#5	99.51	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	SAMPLE#6	97.90	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	DISSOLUTION	97.72	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	STANDARD DEVIATION	1.755	1	sds-1				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	C.V.	1.8	1	%				15545/18- MAY 2002	CAPPUCCIO	MARIANI
	MIN. DISSOLUTION VALUE	95.75	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI
	MAX. DISSOLUTION VALUE	100.05	1	%		80.000		15545/18- MAY 2002	CAPPUCCIO	MARIANI

# PHARMACIA



Global Pharmaceutical Sciences - Italy

## Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 116307  
Sample ID (Request No.) : 20021138  
User sample ID (lot) : IB3G03  
Sample Type: SU010398 (PNU290940AD) CAPSULE  
25MG

### Samples Notes:

Observations on the Method 083GCO1#00, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDS1#01, component: DISSOLUTION

Dissolution at 15 minutes: 96.74% (C.V.= 1.77%)

Request Type: STABILITY  
Stability Study: IB3G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 40°C/75% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

# PHARMACIA



Global Pharmaceutical Sciences - Italy

## Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 116307

Sample ID (Request No.): 20021136

User sample ID (lot): 1B3G03

Sample Type: SU01038 (PNU1290940AD) CAPSULE  
25MG

Request Type: STABILITY

Stability Study: 1B3G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40°C/75% U.R.

Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:

APPROVED

Status:

COMPLETE

### Samples Notes:

Observations on the Method 083GDS01#01, component: DISSOLUTION

Dissolution at 45 minutes: 97.72% (C.V.: 1.86%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.21%

# PHARMACIA



Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 116307  
Sample ID (Request No.): 20021136  
User sample ID (lot): I83G13  
Sample Type: SU010398 (PNU290940AD) CAPSULE  
25MG

### Samples Notes:

Observations on the Method: 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Request Type: STABILITY  
Stability Study: I83G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 40°C/75% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:

**COMPLETE**

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116307  
Sample ID (Request No.): 20021136  
User sample ID (lot): 083G03  
**Sample Type:** SUB0388 (PNU280840AD) CAPSULE  
25MG

Request Type: STABILITY  
Stability Study: 083G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 40°C/75% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

### Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

RRT	Area (%)
-----	----------

# PHARMACIA



Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 116307  
Sample ID (Request No.): 20021136  
User sample ID (Ref): **183G03**  
Sample type: SU010398 (PNU29090AD) CAPSULE  
25MG

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 40°C/75% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**  
Status:  
**COMPLETE**

### Samples Notes:

### Observations on the Method

083GSC01#00, component IMPURITIES NOT LESS THAN 0.05%

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 116207  
Sample ID (Request No.): 20221136

User sample ID (lot): I83G03  
Sample Type: SU010398 (PNU280940AD) CAPSULE  
25MG

Request Type: STABILITY  
Stability Study: I83G03

Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 40°C/75% UR,

Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

Samples Notes:

Observations on the Method      083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.86    0.08

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116207

Sample ID (Request No.): 20021135

User sample ID (lot): 183G03

Sample Type: SU010388 (PNU290940AD) CAPSULE

25MG

Request Type: STABILITY

Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40°C/75% U.R.

Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:

APPROVED

Status:

COMPLETE

### Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.91 0.06

# PHARMACIA



## Analyses Report

Laboratory  
Information  
Management  
System

Global Pharmaceutical Sciences - Italy

Submission Id: 116307

Sample ID (Request No.): 20021136

User sample ID (lot): 183G03

Sample Type: SU010388 (PNU290940AD) CAPSULE  
25MG

Request Type: STABILITY  
Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE.

Storage Condition: 40°C/75% U.R.

Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.94 0.07

Condition:  
**APPROVED**

Status:  
**COMPLETE**

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116307

Sample ID (Request No.): 20021136

User sample ID (lot): **I83G03**

Sample Type: SU010398 (PNU290940AD) CAPSULE  
25MG

Request Type: STABILITY

Stability Study: I83G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40°C/75% U.R.

Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:

APPROVED

Status:

COMPLETE

### Samples Notes:

Observations on the Method      083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

1.00    99.79    (SU11248)

SUBMISSION APPROVED BY      Volker ECK

APPROVE DATE      15-may-2003

SAMPLE APPROVED BY: ROSARIA MARIANI

APPROVE DATE: 17-may-2002

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 116953

Sample ID (Request No.): 20022083

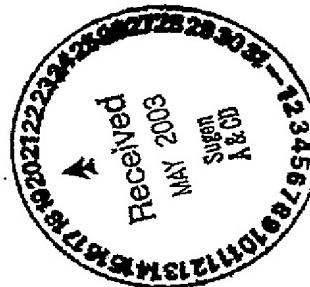
User sample ID (lot): 183G03

### Sample Type: Studio (

23MG

Samples Notes:

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GGC02#00	CAPSULE APPEARANCE	0=UNCHANGED	1	bx				15545/33 AUG2002	CAPPUCIO	FINETTI
	CAPSULE COLOR	0=UNCHANGED	1	bx				15545/33 AUG2002	CAPPUCIO	FINETTI
	CONTENT APPEARANCE	0=UNCHANGED	1	bx				15545/33 AUG2002	CAPPUCIO	FINETTI
	CONTENT COLOR	0=UNCHANGED	1	bx				15545/33 AUG2002	CAPPUCIO	FINETTI
	SMELL	0=UNCHANGED	1	bx				15545/33 AUG2002	CAPPUCIO	FINETTI
083GT01#01_S	(HPLC TITER) SAMPLE#1	97.48	1	%		90.000	- 110.000	15545/33 AUG2002	CAPPUCIO	FINETTI
	(HPLC TITER) SAMPLE#2	96.12	1	%		90.000	- 110.000	15545/33 AUG2002	CAPPUCIO	FINETTI
	(HPLC TITER) SAMPLE#3	97.03	1	%		90.000	- 110.000	15545/33 AUG2002	CAPPUCIO	FINETTI
	AVERAGE TITER (HPLC)	96.88	1	%		90.000	- 110.000	15545/33 AUG2002	CAPPUCIO	FINETTI
083GSC01#00	TOTAL CORRELATED SUBSTANCES	0.32	1	%				15545/33 AUG2002	CAPPUCIO	FINETTI
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	1	%				15545/33 AUG2002	CAPPUCIO	FINETTI
083GKF01#00	(HUMIDITY) SAMPLE #1	2.07	1	%				15545/33 AUG2002	CAPPUCIO	FINETTI
	(HUMIDITY) SAMPLE #2	2.1	1	%				15545/33 AUG2002	CAPPUCIO	FINETTI
	(HUMIDITY) SAMPLE #3	2.2	1	%				15545/33 AUG2002	CAPPUCIO	FINETTI
	AVERAGE HUMIDITY	2.12	1	%				15545/33 AUG2002	CAPPUCIO	FINETTI



Svibpolja.tdf

Printed

20-MAY-2003 01:57 PM by GIUSEPPE MACCARI

Page 1 / 11

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 116953  
Sample ID (Request No.) : 20022083

User sample ID (lot) : IB3G03

Sample Type: SU010388 (PNU290940AD) CAPSULE  
25MG

Request Type: STABILITY  
Stability Study: I83G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 9 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

### Samples Notes:

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#01	SAMPLE#1	89.91	1	%		80.000		15545/33 AUG2002	CAPPUCCIO	FINETTI
	SAMPLE#2	88.29	1	%		80.000		15545/33 AUG2002	CAPPUCCIO	FINETTI
	SAMPLE#3	96.41	1	%		80.000		15545/33 AUG2002	CAPPUCCIO	FINETTI
	SAMPLE#4	96.41	1	%		80.000		15545/33 AUG2002	CAPPUCCIO	FINETTI
	SAMPLE#5	91.00	1	%		80.000		15545/33 AUG2002	CAPPUCCIO	FINETTI
	SAMPLE#6	91.00	1	%		80.000		15545/33 AUG2002	CAPPUCCIO	FINETTI
	DISSOLUTION	92.17	1	%		80.000		15545/33 AUG2002	CAPPUCCIO	FINETTI
	STANDARD DEVIATION	3.431	1	std-1				15545/33 AUG2002	CAPPUCCIO	FINETTI
	C.V.	3.72	1	%				15545/33 AUG2002	CAPPUCCIO	FINETTI
	MIN. DISSOLUTION VALUE	88.29	1	%		80.000		15545/33 AUG2002	CAPPUCCIO	FINETTI
	MAX. DISSOLUTION VALUE	96.41	1	%		80.000		15545/33 AUG2002	CAPPUCCIO	FINETTI

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 116953  
Sample ID (Request No.): 20022083  
User sample ID (loc): 1B3G03  
Sample Type: SU010398 (PNU290940AD) CAPSULE  
25MG

Samples Notes:

Observations on the Method

083GC002#00, component: CAPSULE APPEARANCE  
Opaque brick red colored, format 3, hard gelatin capsule

Request Type: STABILITY  
Stability Study: 1B3G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 9 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 116853  
Sample ID (Request No.): 20022083

User sample ID (lot): **IB3G03**

Sample Type: SU010398 (PNU290940AD) CAPSULE  
25MG

Samples Notes:

Observations on the Method 083GCO02#00, component: CAPSULE APPEARANCE  
containing yellow-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION  
Average dissolution at 15 minutes: 74.93% (interval 63.91%-83.97%)

Condition: APPROVED  
Status: COMPLETE

Request Type: STABILITY  
Stability Study: IB3G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 9 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116953

Sample ID (Request No.): 20022083

User sample ID (lot): B3G03

Sample Type: SU010398 (PNU290940AD) CAPSULE

25MG

### Samples Notes:

Observations on the Method 083GD501401, component: DISSOLUTION

Average dissolution at 45 minutes: 96.56% (interval 95.33%-98.04%)

Observations on the Method 083GS501400, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.32%

Request Type: STABILITY  
Stability Study: B3G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 9 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116953

Sample ID (Request No.) 20022003

User sample ID (lot) 183G03

Sample Type: SU010398 (PNU280840AD) CAPSULE

25MG

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 9 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Condition:  
**APPROVED**

Status:

**COMPLETE**

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116953  
Sample ID (Request No.) 20022083  
User sample ID (lot) I83G03  
Sample Type: SU010398 (PNU280940AD) CAPSULE  
26MG

Observations on the Method

Request Type: STABILITY

Stability Study: I83G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:

**APPROVED**

Status:

**COMPLETE**

Samples Notes:

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

RRT AREA%

svfbolta.rdf

Printed 20-MAY-2003 01:57 PM by GIUSEPPE MACCARI

Page 7 / 11

# PHARMACIA



## Analyses Report

Laboratory  
Information  
Management  
System

Global Pharmaceutical Sciences - Italy

SubmissionId: 116953

Sample ID (Request No.): 20022083

User sample ID (lot): **1B3G03**

Sample Type: SU010398 (PNU290840AD) CAPSULE  
25MG

Request Type: STABILITY

Stability Study: 1B3G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

### Samples Notes:

Observations on the Method      083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.66

0.11

# PHARMACIA

Global Pharmaceutical Sciences - Italy



## Analyses Report

Laboratory  
Information  
Management  
System

Submission id: 116953  
Sample ID (Request No.) 20022083

User sample ID (lot) 183G03  
Sample Type: SU010338 (PNU290940AD) CAPSULE

25MG

Request Type: STABILITY  
Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.

Time Label: 9 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

### Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.94 0.05

# PHARMACIA



Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 116953  
Sample ID (Request No.) 20022003

User sample ID (lot) 183G03  
Sample Type: SLU01039B (PNU290940AD) CAPSULE

25MG

### Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

1.00 99.88 (SU11248)

Condition:  
**APPROVED**

Status :

**COMPLETE**

Request Type: STABILITY

Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 116653  
Sample ID (Request No.) 20322033  
User Sample ID (lot) I83G03  
Sample Type: SU010398 (PNU28090AD) CAPSULE  
25MG

Samples Notes:

Observations on the Method

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

1.06    0.16

Request Type: STABILITY

Stability Study: I83G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:

APPROVED

Status:

COMPLETE

SUBMISSION APPROVED BY Volker ECK

APPROVE DATE 15-may-2003

SAMPLE APPROVED BY: GIORGIO FINETTI

APPROVE DATE: 08-sep-2002

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 117497

Sample ID (Request No.): 200222972

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU280940AD) CAPSULE  
25MG

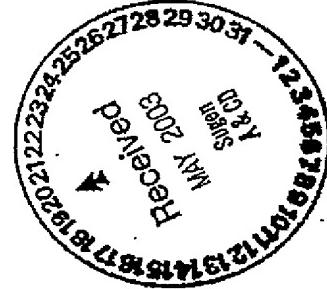
Samples Notes:

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 20°C/60% R.H.  
Time Label: 12 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCO02#00	CAPSULE APPEARANCE	0=UNCHANGED	1	txt				15545/59- NOV 2002	CAPPUCIO	MACCARI
	CAPSULE COLOR	0=UNCHANGED	1	txt				15545/59- NOV 2002	CAPPUCIO	MACCARI
	CONTENT APPEARANCE	0=UNCHANGED	1	txt				15545/59- NOV 2002	CAPPUCIO	MACCARI
	CONTENT COLOR	0=UNCHANGED	1	txt				15545/59- NOV 2002	CAPPUCIO	MACCARI
	SMELL	0=UNCHANGED	1	txt				15545/59- NOV 2002	CAPPUCIO	MACCARI
083GT01#01_S	(HPLC TITER) SAMPLE#1	95.84	1	%		90.000	- 110.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	(HPLC TITER) SAMPLE#2	96.17	1	%		90.000	- 110.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	(HPLC TITER) SAMPLE#3	97.03	1	%		90.000	- 110.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	AVERAGE TITER (HPLC)	96.35	1	%		90.000	- 110.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
083GSC01#00	TOTAL CORRELATED SUBSTANCES	0.15	1	%				15545/59- NOV 2002	CAPPUCIO	MACCARI
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	1	%				15545/59- NOV 2002	CAPPUCIO	MACCARI
083GK#01#00	(HUMIDITY) SAMPLE #1	2.1	1	%				15545/59- NOV 2002	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #2	2.2	1	%				15545/59- NOV 2002	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #3	2.1	1	%				15545/59- NOV 2002	CAPPUCIO	MACCARI
	AVERAGE HUMIDITY	2.13	1	%				15545/59- NOV 2002	CAPPUCIO	MACCARI



# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 117497

Sample ID (Request No.): 20022972

User sample ID (lot): **IB3G03**

Sample Type: SU010398 (PNU290940AD) CAPSULE  
25MG

Request Type: STABILITY

Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 12 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:

**APPROVED**

Status:

**COMPLETE**

### Samples Notes:

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#01	SAMPLE#1	88.22	1	%		80.000	100.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	SAMPLE#2	93.41	1	%		80.000	100.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	SAMPLE#3	101.19	1	%		80.000	100.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	SAMPLE#4	97.04	1	%		80.000	100.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	SAMPLE#5	94.97	1	%		80.000	100.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	SAMPLE#6	92.89	1	%		80.000	100.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	DISSOLUTION	94.62	1	%		80.000	100.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	STANDARD DEVIATION	4.349	1	std-1				15545/59- NOV 2002	CAPPUCIO	MACCARI
	C.V.	4.6	1	%				15545/59- NOV 2002	CAPPUCIO	MACCARI
	MIN. DISSOLUTION VALUE	88.22	1	%		80.000	100.000	15545/59- NOV 2002	CAPPUCIO	MACCARI
	MAX. DISSOLUTION VALUE	101.19	1	%		80.000	100.000	15545/59- NOV 2002	CAPPUCIO	MACCARI

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 117497

Sample ID (Request No.) : 20022972

User sample ID (lot) : 1B3G03

Sample Type: SU010398 (PNL250940AD) CAPSULE  
25MG

Samples Notes:

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GMC01#01 TOTAL AEROBIC BACTERIA .1 IN LIMITS										
						ABSENT		15545/59- NOV 2002	CAPPUCIO	MACCARI
						IN LIMITS				
						NOT DETECTABLE				
						NOT PLANNED				
ESCHERICHIA COLI	ABSENT	1	txt			ABSENT		15545/59- NOV 2002	CAPPUCIO	MACCARI
						NOT DETECTABLE				
						NOT PLANNED				
SALMONELLA	ABSENT	1	txt			ABSENT		15545/59- NOV 2002	CAPPUCIO	MACCARI
						NOT DETECTABLE				
						NOT PLANNED				
FUNGI	IN LIMITS	1	txt			ABSENT		15545/59- NOV 2002	CAPPUCIO	MACCARI
						IN LIMITS				
						NOT DETECTABLE				
						NOT PLANNED				
STAPHYLOCOCCUS AUREUS	ABSENT	1	txt			ABSENT		15545/59- NOV 2002	CAPPUCIO	MACCARI
						NOT DETECTABLE				
						NOT PLANNED				
ENTEROBACTERIA	IN LIMITS	1	txt			ABSENT		15545/59- NOV 2002	CAPPUCIO	MACCARI
						IN LIMITS				
						NOT DETECTABLE				

Condition:  
**APPROVED**

Status:

**COMPLETE**

Preparation purpose: CLINICAL SUPPLY + STABILITY

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 117497

Sample ID (Request No.): 20022972

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU280940AD) CAPSULE  
25MG

Samples Notes:

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
--------	----------	---------	-------	------	-------	-----------	-----------	--------------------	---------	----------

083GMC01#01 ENTEROBACTERIA

IN LIMITS

1

b1

NOT PLANNED

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 12 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

status:

**COMPLETE**

# PHARMACIA

Global Pharmaceutical Sciences - Italy

## Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 117497

Sample ID (Request No.): 20022972

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU28090AD) CAPSULE  
25MG

### Samples Notes:

#### Observations on the Method

083G02800, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsula containing yellow-orange powder

#### Observations on the Method

083GDS01#01, component: DISSOLUTION

Average dissolution at 15 minutes: 77.24% (C.V. 7.27%)

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/80% U.R.  
Time Label: 12 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

# PHARMACIA

Laboratory  
Information  
Management  
System

## Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 117497

Sample ID (Request No.): 200222972

User sample ID (lot): **I83G03**

Sample Type: SU010398 (PNU290940AD) CAPSULE

25MG

Request Type: STABILITY

Stability Study: I83G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 12 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:

APPROVED

Status:

COMPLETE

### Samples Notes:

Observations on the Method 083GDS0#01, component: DISSOLUTION

Average dissolution 45 minutes: 97.39% (C.V.: 2.25%)

Observations on the Method 083GMC0#01, component: TOTAL AEROBIC BACTERIA

Total bacterial count < 10 Ufc/g (Report num. 02.16971)

Observations on the Method 083GMC0#01, component: ENTEROBACTERIA

Enterobacteria < 10 Ufc/g (Report num. 02.16971)

Observations on the Method 083GMC0#01, component: ESCHERICHIA COLI

Report num. 02.16971

Observations on the Method 083GMC0#01, component: FUNGI

Fungi (Yeast + Molds) < 10 Ufc/g (Report num. 02.16971)

Observations on the Method 083GMC0#01, component: SALMONELLA

Report num. 02.16971

Observations on the Method 083GMC0#01, component: STAPHYLOCOCCUS AUREUS

Report num. 02.16971

Observations on the Method 083GSC0#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.15%

# PHARMACIA

Global Pharmaceutical Sciences - Italy



## Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 117497  
Sample ID (Request No.): 20022992  
User sample ID (lot): 183G03  
Sample Type: SU010398 (PNU29090AD) CAPSULE  
25MG

Request Type: STABILITY

Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 12 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:

**COMPLETE**

### Samples Notes:

Observations on the Method      083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 117497  
Sample ID /Request No.: 20022972  
User sample ID /lot#: 183G03  
Sample Type: SU010398 (PNU290940AD) CAPSULE  
25MG

Samples Notes:

Observations on the Method: 083GC0#00, component: IMPURITIES NOT LESS THAN 0.05%

RRT

Area (%)

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 12 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 117497  
Sample ID (Request No.): 20022972

User sample ID (lot): IR3G03  
Sample Type: SU010388 (PNU299940AD) CAPSULE

25MG

Samples Notes:

Observations on the Method 083GC0#00, component: IMPURITIES NOT LESS THAN 0.05%

Condition:  
**APPROVED**

Status:  
**COMPLETE**

---

Request Type: STABILITY  
Stability Study: 083G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 12 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

# PHARMACIA

Global Pharmaceutical Sciences - Italy



## Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 117497  
Sample ID (Request No.) 200222972

User sample ID (lot) B3G03

Sample Type: SU010398 (PNU290940AD) CAPSULE  
25MG

### Samples Notes:

Observations on the Method  
083 GC01#00, component: IMPURITIES NOT LESS  
THAN 0.05%

0.65      0.10

Request Type: STABILITY  
Stability Study: B3G03

Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.

Time Label: 12 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

# PHARMACIA

Global Pharmaceutical Sciences - Italy

## Analyses Report

Laboratory  
Information  
Management  
System

Submission Id: 117497  
Sample ID (Request No.) 20022872

User sample ID (lot) 183G03

Sample Type: SU01039B (PNU250940AD) CAPSULE  
25MG

### Samples Notes:

Observations on the Method 083GC01#00, component: IMPURITIES NOT LESS  
THAN 0.05%

0.94      0.05

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 12 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

# PHARMACIA

Global Pharmaceutical Sciences - Italy

Laboratory  
Information  
Management  
System

## Analyses Report

Submission Id: 117497

Sample ID (Request No.): 20022972

User sample ID (lot): 183G03

Sample Type: SU010388 (PNU290940AD) CAPSULE

25MG.

Samples Notes:

Observations on the Method  
083GC0#00, component: IMPURITIES NOT LESS  
THAN 0.05%

1.00 99.85 (SU 1/1248)

Request Type: STABILITY  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/80% U.R.  
Time Label: 12 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition :  
**APPROVED**

Status :  
**COMPLETE**

SUBMISSION APPROVED BY Volker ECK  
APPROVE DATE 16-may-2003

SAMPLE APPROVED BY : GIUSEPPE MACCARI  
APPROVE DATE : 27-nov-2002

Time Points

# PHARMACIA

Laboratory  
Information  
Management  
System

## Pharmaceutical Development

Submission Id: 116953

Sample ID (Request No.): 20022063

User sample ID (lot): 183G03

Sample Date : 18/07/2002

Sample Type: SUB10398 (PNU290340AD) CAPSULE

25MG

Stability Study Start Date : 18/10/2001

Stability Protocol Number: 2001017 - Manufacturer : PD/Pharmacia Neniano

### Samples Notes:

Batch Manufacture Date : 17/09/2001

Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

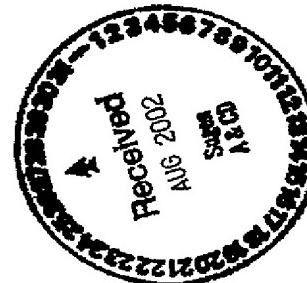
Condition:  
**APPROVED**

Status:

COMPLETE

## Integrated Analytical Bulletin

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier	
083GC02#0	CAPSULE APPEARANCE	0=UNCHANGED	txt					15545/32 AUG2002	CAPPUCIO	FINETTI	
	CAPSULE COLOR	0=UNCHANGED	txt					15545/32 AUG2002	CAPPUCIO	FINETTI	
	CONTENT APPEARANCE	0=UNCHANGED	txt					15545/32 AUG2002	CAPPUCIO	FINETTI	
	CONTENT COLOR	0=UNCHANGED	txt					15545/32 AUG2002	CAPPUCIO	FINETTI	
	SMELL	0=UNCHANGED	txt					15545/32 AUG2002	CAPPUCIO	FINETTI	
083GCT101#01_S	(HPLC TITER) SAMPLE#1	97.48	%			90.000	-	110.000	15545/32 AUG2002	CAPPUCIO	FINETTI
	(HPLC TITER) SAMPLE#2	96.12	%			90.000	-	110.000	15545/32 AUG2002	CAPPUCIO	FINETTI
	(HPLC TITER) SAMPLE#3	97.03	%			90.000	-	110.000	15545/32 AUG2002	CAPPUCIO	FINETTI
	AVERAGE TITER (HPLC)	96.88	%			90.000	-	110.000	15545/32 AUG2002	CAPPUCIO	FINETTI
083GSC01#0	TOTAL CORRELATED SUBSTANCES	0.32	%					15545/32 AUG2002	CAPPUCIO	FINETTI	
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	%					15545/32 AUG2002	CAPPUCIO	FINETTI	
083GKF01#00	(HUMIDITY) SAMPLE #1	2.07	%					15545/32 AUG2002	CAPPUCIO	FINETTI	
	(HUMIDITY) SAMPLE #2	2.1	%					15545/32 AUG2002	CAPPUCIO	FINETTI	
	(HUMIDITY) SAMPLE #3	2.2	%					15545/32 AUG2002	CAPPUCIO	FINETTI	
	AVERAGE HUMIDITY	2.12	%					15545/32 AUG2002	CAPPUCIO	FINETTI	



# PHARMACIA

Laboratory  
Information  
Management  
System

## Integrated Analytical Bulletin

### Pharmaceutical Development



Submission Id: 116953

Sample ID (Request No.): 20022003

User sample ID (lot): 183G03

Sample Date : 18/07/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE

25MG

Stability Study Start Date : 18/10/2001

Stability Protocol Number : 2001017 - Manufacturer : PDI/Pharmacia Neri/ano

#### Samples Notes:

Batch Manufacture Date : 17/09/2001  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 9 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition :  
**APPROVED**  
Status :  
**COMPLETE**

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083G0301#0	SAMPLE#1	89.91	%			80.000		15545/32 AUG2002	CAPPUCIO	FINETTI
1	SAMPLE#2	88.29	%			80.000		15545/32 AUG2002	CAPPUCIO	FINETTI
	SAMPLE#3	96.41	%			80.000		15545/32 AUG2002	CAPPUCIO	FINETTI
	SAMPLE#4	96.41	%			80.000		15545/32 AUG2002	CAPPUCIO	FINETTI
	SAMPLE#5	91.00	%			80.000		15545/32 AUG2002	CAPPUCIO	FINETTI
	SAMPLE#6	91.00	%			80.000		15545/32 AUG2002	CAPPUCIO	FINETTI
	DISSOLUTION	92.17	%			80.000		15545/32 AUG2002	CAPPUCIO	FINETTI
	STANDARD DEVIATION	3.431			std-1			15545/32 AUG2002	CAPPUCIO	FINETTI
	C.V.	3.72	%					15545/32 AUG2002	CAPPUCIO	FINETTI
	MIN. DISSOLUTION VALUE	88.29	%			80.000		15545/32 AUG2002	CAPPUCIO	FINETTI
	MAX. DISSOLUTION VALUE	96.41	%			80.000		15545/32 AUG2002	CAPPUCIO	FINETTI

# PHARMACIA

Laboratory  
Information  
Management  
System

## Integrated Analytical Bulletin

### Pharmaceutical Development

Submission Id: 116953

Sample ID (Request No.): 20022083  
User sample ID (lot): 1B3G03

Sample Date: 18/07/2002

Sample Type: SU010398 (PNU28094QAD) CAPSULE  
25MG  
Stability Protocol Number : 2001011 - Manufacturer : PDI/Pharmacia Nerviano

#### Samples Notes:

Observations on the Method 083GC002#00, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule containing red-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

Average dissolution at 15 minutes: 74.93% (interval 63.91%-89.37%)

Average dissolution at 45 minutes: 96.59% (interval 95.33%-98.04%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.32%

RRT	AREA%
0.66	0.11
0.94	0.05
1.00	99.68 (SLU1246)
1.06	0.16

Approved by: FINETTI  
Approval date: 05-aug-2002

# PHARMACIA

Laboratory  
Information  
Management  
System

## Pharmaceutical Development

Submission Id: 116305

Sample ID (Request No.): 2002/134

User sample ID (lot): 183G03

Sample Date: 18/04/2002

Sample Type: SU010388 (PNU290940AD) CAPSULE

25MG

Stability Study Start Date: 18/10/2001

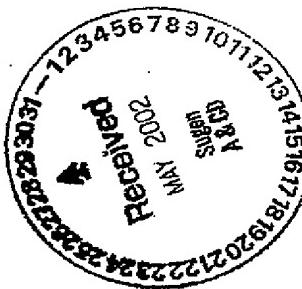
Stability Protocol Number: 20010177 - Manufacturer: PDI/Pharmacia Nenjano

Samples Notes:

Condition: APPROVED  
Batch Manufacture Date: 17/09/2001  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

## Integrated Analytical Bulletin

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCO02#0	CAPSULE APPEARANCE	0=UNCHANGED	txt				15545/18- MAY 2002	CAPPUCIO	MARIANI
0	CAPSULE COLOR	0=UNCHANGED	txt				15545/18- MAY 2002	CAPPUCIO	MARIANI
	CONTENT APPEARANCE	0=UNCHANGED	txt				15545/18- MAY 2002	CAPPUCIO	MARIANI
	CONTENT COLOR	0=UNCHANGED	txt				15545/18- MAY 2002	CAPPUCIO	MARIANI
	SMELL	0=UNCHANGED	txt				15545/18- MAY 2002	CAPPUCIO	MARIANI
083GT01#01	(HPLC TITER) SAMPLE#1	97.95	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
_S	(HPLC TITER) SAMPLE#2	97.96	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
	(HPLC TITER) SAMPLE#3	97.25	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
	AVERAGE TITER (HPLC)	97.72	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
083GSC01#0	TOTAL CORRELATED SUBSTANCES 0.18	%					15545/18- MAY 2002	CAPPUCIO	MARIANI
0	IMPURITIES NOT LESS THAN 0.05% SEE TEXT	%					15545/18- MAY 2002	CAPPUCIO	MARIANI
083GKF01#00	(HUMIDITY) SAMPLE #1	2.37	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	(HUMIDITY) SAMPLE #2	2.4	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	(HUMIDITY) SAMPLE #3	2.38	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	AVERAGE HUMIDITY	2.38	%				15545/18- MAY 2002	CAPPUCIO	MARIANI



Printed 17-MAY-2002 05:05 PM by MARIANI

symbol/integrated.rdf

Page 1 / 3

# PHARMACIA

## Pharmaceutical Development

Laboratory  
Information  
Management  
System

### Integrated Analytical Bulletin

Submission Id: 116305

Sample ID (Request No.): 20021134

User sample ID (lot): 183G03

Sample Date: 18/04/2002

Sample Type: SLIC010598 (PNU290940AD) CAPSULE

25MG

Stability Study Start Date: 18/10/2001

Stability Protocol Number: 2003/017 - Manufacturer: PD/Pharmacia Nerviano

#### Samples Notes:

Batch Manufacture Date: 17/09/2001

Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#01	SAMPLE#1	94.01	%		80.000		15545/18-MAY 2002	CAPPUCIO	MARIANI
	SAMPLE#2	99.04	%		80.000		15545/18-MAY 2002	CAPPUCIO	MARIANI
	SAMPLE#3	100.16	%		80.000		15545/18-MAY 2002	CAPPUCIO	MARIANI
	SAMPLE#4	94.01	%		80.000		15545/18-MAY 2002	CAPPUCIO	MARIANI
	SAMPLE#5	88.41	%		80.000		15545/18-MAY 2002	CAPPUCIO	MARIANI
	SAMPLE#6	94.56	%		80.000		15545/18-MAY 2002	CAPPUCIO	MARIANI
	DISSOLUTION	95.03	%	std-1			15545/18-MAY 2002	CAPPUCIO	MARIANI
	STANDARD DEVIATION	4.208					15545/18-MAY 2002	CAPPUCIO	MARIANI
	C.V.	4.43	%				15545/18-MAY 2002	CAPPUCIO	MARIANI
	MIN. DISSOLUTION VALUE	88.41	%		80.000		15545/18-MAY 2002	CAPPUCIO	MARIANI
	MAX. DISSOLUTION VALUE	100.16	%		80.000		15545/18-MAY 2002	CAPPUCIO	MARIANI

# PHARMACIA

## Pharmaceutical Development

Submission Id: 116305  
Sample ID (Request No.) 20021134

User Sample ID (lot) 183G03

Sample Date : 18/04/2002

Sample Type: SU010398 (PNU280940AD) CAPSULE

25MG

Stability Study Start Date : 18/10/2001

Stability Protocol Number : 2001017 - Manufacturer : PD/Pharmacia Nerviano

### Samples Notes:

Observations on the Method 081GCO02400, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule containing red-orange powder

Observations on the Method 081GDS01#01, component: DISSOLUTION

Dissolution at 15 minutes: 76.5 % (C.V. = 13.9%)

Dissolution at 45 minutes: 99.97% (C.V. = 1.16%)

Observations on the Method 081GSC01#00, component: IMPURITIES NOT LESS

THAN 0.05%

Total correlated substances: 0.18%

RRT	Area (%)
0.66	0.12
0.91	0.06
1.00	99.82 (SU11248)

Approved by: MARIANI  
Approval date: 17-may-2002

# PHARMACIA

## Pharmaceutical Development

Laboratory  
Information  
Management  
System

### Integrated Analytical Bulletin

Submission Id: 116307  
Sample ID (Request No.): 20021136

User sample ID (lot): **1B3G03**

Sample Date : 18/04/2002

Sample Type: SU010398 (PNU280840AD) CAPSULE

25MG

Stability Study Start Date : 18/01/2001

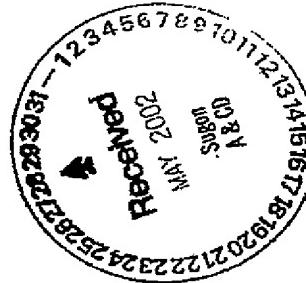
Stability Protocol Number : 2001017 - Manufacturer : PDI/Pharmacia Nerviano

#### Samples Notes:

Batch Manufacture Date : 17/09/2001  
Stability Study: (88G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 40°C/75% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**  
Status:  
**COMPLETE**

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCC02#0	CAPSULE APPEARANCE	0=UNCHANGED	txt				15545/18- MAY 2002	CAPPUCIO	MARIANI
0	CAPSULE COLOR	0=UNCHANGED	txt				15545/18- MAY 2002	CAPPUCIO	MARIANI
	CONTENT APPEARANCE	0=UNCHANGED	txt				15545/18- MAY 2002	CAPPUCIO	MARIANI
	CONTENT COLOR	0=UNCHANGED	txt				15545/18- MAY 2002	CAPPUCIO	MARIANI
	SMELL	0=UNCHANGED	txt				15545/18- MAY 2002	CAPPUCIO	MARIANI
083GTI1#01	(HPLC TITER) SAMPLE#1	97.43	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
S	(HPLC TITER) SAMPLE#2	98.55	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
	(HPLC TITER) SAMPLE#3	97.23	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
	AVERAGE TITER (HPLC)	97.74	%		90.000	- 110.000	15545/18- MAY 2002	CAPPUCIO	MARIANI
083GSC01#0	TOTAL CORRELATED SUBSTANCES	0.21	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
0	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
083GKF01#00	(HUMIDITY) SAMPLE #1	2.77	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	(HUMIDITY) SAMPLE #2	2.9	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	(HUMIDITY) SAMPLE #3	2.8	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	AVERAGE HUMIDITY	2.82	%				15545/18- MAY 2002	CAPPUCIO	MARIANI



# PHARMACIA

Laboratory  
Information  
Management  
System

## Pharmaceutical Development

Submission Id: 116307

Sample ID [Request No.]: 200211136

User sample ID (lot): 183G03

Sample Date : 18/04/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE  
25MG

Stability Study Start Date : 18/10/2001

Stability Protocol Number : 2001017 - Manufacturer : Pd/Pharmacia Novartis

Samples Notes:

Batch Manufacture Date : 17/09/2001  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 40°C/75% U.R.  
Time Label: 6 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#0	SAMPLE#1	98.29	%		80.000		15545/18- MAY 2002	CAPPUCIO	MARIANI
1	SAMPLE#2	95.75	%		80.000		15545/18- MAY 2002	CAPPUCIO	MARIANI
	SAMPLE#3	96.82	%		80.000		15545/18- MAY 2002	CAPPUCIO	MARIANI
	SAMPLE#4	100.05	%		80.000		15545/18- MAY 2002	CAPPUCIO	MARIANI
	SAMPLE#5	99.51	%		80.000		15545/18- MAY 2002	CAPPUCIO	MARIANI
	SAMPLE#6	97.90	%		80.000		15545/18- MAY 2002	CAPPUCIO	MARIANI
	DISSOLUTION	97.72	%		80.000		15545/18- MAY 2002	CAPPUCIO	MARIANI
	STANDARD DEVIATION	1.755		std-1			15545/18- MAY 2002	CAPPUCIO	MARIANI
	C.V.	1.8	%				15545/18- MAY 2002	CAPPUCIO	MARIANI
	MIN. DISSOLUTION VALUE	95.75	%		80.000		15545/18- MAY 2002	CAPPUCIO	MARIANI
	MAX. DISSOLUTION VALUE	100.05	%		80.000		15545/18- MAY 2002	CAPPUCIO	MARIANI

# PHARMACIA

Laboratory  
Information  
Management  
System

## Integrated Analytical Bulletin

### Pharmaceutical Development

Submission Id: 116307

Sample ID (Request No.): 20021136

User sample ID (lot): 1B3G03

Sample Date : 18/04/2002

Sample Type: SU010398 (PNU280940AD) CAPSULE

25MG

Stability Study Start Date : 18/10/2001

Stability Protocol Number : 2001017 - Manufacturer : PDP/Pharmacia Nerviano

#### Samples Notes:

Batch Manufacture Date : 17/09/2001

Stability Study: 1B3G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40°C/75% U.R.

Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

Approved by: MARIANI  
Approval date: 17-may-2002

#### Observations on the Method 083GCO02#00, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule containing red-orange powder

#### Observations on the Method 083GDS01#01, component: DISSOLUTION

Dissolution at 15 minutes: 96.74% (C.V. = 1.77%)

Dissolution at 45 minutes: 97.72% (C.V. = 1.88%)

#### Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.21%

RRT	Area (%)
0.66	0.08
0.91	0.06
0.94	0.07
1.00	98.79 (SU11248)

# PHARMACIA

## Pharmaceutical Development



Laboratory  
Information  
Management  
System

## Integrated Analytical Bulletin

Submission Id: 115471  
Sample ID (Request No.): 20020165  
User sample ID (lot): 183G03

Sample Date : 18/01/2002

Sample Type: SU010388 (PNU290940AD) CAPSULE

25MG

Stability Study Start Date : 18/10/2001

Stability Protocol Number : 12001017 - Manufacturer : PDI/Pharmacia Nerviano

### Samples Notes:

Batch Manufacture Date : 17/09/2001  
Request Type: RELEASE  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 3 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCO02H0_0	CAPSULE APPEARANCE	0=UNCHANGED	txt				143/44- JAN 2002	CAPPUCIO	MACCARI
	CAPSULE COLOR	0=UNCHANGED	txt				143/44- JAN 2002	CAPPUCIO	MACCARI
	CONTENT APPEARANCE	0=UNCHANGED	txt				143/44- JAN 2002	CAPPUCIO	MACCARI
	CONTENT COLOR	0=UNCHANGED	txt				143/44- JAN 2002	CAPPUCIO	MACCARI
	SMELL	0=UNCHANGED	txt				143/44- JAN 2002	CAPPUCIO	MACCARI
083GTT01#01_S	(HPLC TITER) SAMPLE#1	98.84	%		90.000	- 110.000	143/44- JAN 2002	CAPPUCIO	MACCARI
	(HPLC TITER) SAMPLE#2	96.31	%		90.000	- 110.000	143/44- JAN 2002	CAPPUCIO	MACCARI
	(HPLC TITER) SAMPLE#3	96.41	%		90.000	- 110.000	143/44- JAN 2002	CAPPUCIO	MACCARI
	AVERAGE TITER (HPLC)	97.19	%		90.000	- 110.000	143/44- JAN 2002	CAPPUCIO	MACCARI
083GSC01#00_0	TOTAL CORRELATED SUBSTANCES 0.21	%					143/44- JAN 2002	CAPPUCIO	MACCARI
	IMPURITIES NOT LESS THAN 0.05% SEE TEXT	%					143/44- JAN 2002	CAPPUCIO	MACCARI
083GKF01#00	(HUMIDITY) SAMPLE #1	1.9	%				143/44- JAN 2002	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #2	1.82	%				143/44- JAN 2002	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #3	1.9	%				143/44- JAN 2002	CAPPUCIO	MACCARI
	AVERAGE HUMIDITY	1.87	%				143/44- JAN 2002	CAPPUCIO	MACCARI

svfbolointegrated.rdf

Printed 20-MAR-2002 05:46 PM by MARIANI

Page 1 / 3

# PHARMACIA

## Pharmaceutical Development

Laboratory  
Information  
Management  
System

### Integrated Analytical Bulletin

Submission Id: 115471

Sample ID (Request No.): 20020165

User sample ID (lot): 183G03

Sample Date : 18/01/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE

25MG

Stability Study Start Date : 18/10/2001

Stability Protocol Number : 2001017 - Manufacturer : PD/Pharmacia Nerviano

Samples Notes:

Condition: APPROVED  
Batch Manufacture Date : 17/08/2001  
Request Type: RELEASE  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/60% U.R.  
Time Label: 3 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GD501#0	SAMPLE#1	101.14	%	80.000	143/44-	JAN 2002	CAPPUCIO	MACCARI	
1	SAMPLE#2	106.48	%	80.000	143/44-	JAN 2002	CAPPUCIO	MACCARI	
	SAMPLE#3	109.97	%	80.000	143/44-	JAN 2002	CAPPUCIO	MACCARI	
	SAMPLE#4	106.90	%	80.000	143/44-	JAN 2002	CAPPUCIO	MACCARI	
	SAMPLE#5	105.33	%	80.000	149/44-	JAN 2002	CAPPUCIO	MACCARI	
	SAMPLE#6	104.08	%	80.000	143/44-	JAN 2002	CAPPUCIO	MACCARI	
	DISSOLUTION	105.65	%	80.000	143/44-	JAN 2002	CAPPUCIO	MACCARI	
	STANDARD DEVIATION	2.96	std <sup>-1</sup>		143/44-	JAN 2002	CAPPUCIO	MACCARI	
	C.V.	2.8	%		143/44-	JAN 2002	CAPPUCIO	MACCARI	
	MIN. DISSOLUTION VALUE	101.14	%	80.000	143/44-	JAN 2002	CAPPUCIO	MACCARI	
	MAX. DISSOLUTION VALUE	109.97	%	80.000	143/44-	JAN 2002	CAPPUCIO	MACCARI	

# PHARMACIA

## Pharmaceutical Development

Laboratory  
Information  
Management  
System

### Integrated Analytical Bulletin

Submission Id: 115471

Sample ID (Request No.): 20020165

User sample ID (lot): 1B3G03

Sample Date : 18/01/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE

25MG

Stability Study Start Date : 18/10/2001

Stability Protocol Number : 2001017 - Manufacturer : PDI/Pharmacia Nerviano

#### Samples Notes:

#### Observations on the Method

083GCC02400, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule containing yellow-orange powder

#### Observations on the Method

083GDS01#01, component: DISSOLUTION

#### Observations on the Method

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.21%

#### RRT

#### Area (%)

1.00	99.79	(SU 11248)
1.97	0.09	
2.06	0.12	

Approved by: MACCARI

Approval date: 04-feb-2002

# PHARMACIA

Laboratory  
Information  
Management  
System

## Pharmaceutical Development

Submission Id: 115673

Sample ID (Request No.): 20020167

User sample ID (lot): 183G03

Sample Date : 18/01/2002

Sample Type: SU010398 (PNU290940AD)CAPSULE

25MG

Stability Study Start Date : 18/10/2001

Stability Protocol Number : 20010117 - Manufacturer : PD/Pharmacia Nerviano

### Samples Notes:

Batch Manufacture Date : 17/09/2001  
Request Type: RELEASE  
Stability Study: 183G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 40°C/75% U.R.  
Time Label: 3 MONTHS  
Preparation purpose: CLINICAL SUPPLY + STABILITY

## Integrated Analytical Bulletin

Condition:  
**APPROVED**

Status:  
**COMPLETE**

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCO02#0	CAPSULE APPEARANCE	0=UNCHANGED	txt				143/44- JAN 2002	CAPPUCIO	MACCARI
0	CAPSULE COLOR	0=UNCHANGED	txt				143/44- JAN 2002	CAPPUCIO	MACCARI
	CONTENT APPEARANCE	0=UNCHANGED	txt				143/44- JAN 2002	CAPPUCIO	MACCARI
	CONTENT COLOR	0=UNCHANGED	txt				143/44- JAN 2002	CAPPUCIO	MACCARI
	SMELL	0=UNCHANGED	txt				143/44- JAN 2002	CAPPUCIO	MACCARI
083GT01#01	(HPLC TITER) SAMPLE#1	99.21	%		90.000	- 110.000	143/44-JAN 2002	CAPPUCIO	MACCARI
_S	(HPLC TITER) SAMPLE#2	96.18	%		90.000	- 110.000	143/44-JAN 2002	CAPPUCIO	MACCARI
	(HPLC TITER) SAMPLE#3	95.82	%		90.000	- 110.000	143/44-JAN 2002	CAPPUCIO	MACCARI
	AVERAGE TITER (HPLC)	97.07	%		90.000	- 110.000	143/44-JAN 2002	CAPPUCIO	MACCARI
083GSC01#0	TOTAL CORRELATED SUBSTANCES	0.41	%				143/44- JAN 2002	CAPPUCIO	MACCARI
0	IMPURITIES NOT LESS THAN 0.05% SEE TEXT	%					143/44- JAN 2002	CAPPUCIO	MACCARI
083GKF01#00	(HUMIDITY) SAMPLE #1	1.8	%				143/44- JAN 2002	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #2	1.7	%				143/44- JAN 2002	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #3	1.9	%				143/44- JAN 2002	CAPPUCIO	MACCARI
	AVERAGE HUMIDITY	1.8	%				143/44- JAN 2002	CAPPUCIO	MACCARI

# PHARMACIA

Laboratory  
Information  
Management  
System

## Pharmaceutical Development

Submission Id: 115473

Sample ID (Request No.): 20020167

User sample ID (lot): 1B3G03

Sample Date: 18/01/2002

Sample Type: SU010398 (PNW1290840AD) CAPSULE

25MG

Stability Study Start Date: 18/10/2001

Stability Protocol Number: 2301017 - Manufacturer: PDI/Pharmacia Nerviano

Samples Notes:

Batch Manufacture Date : 17/09/2001

Request Type: RELEASE

Stability Study: 1B3G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40°C/75% U.R.

Time Label: 3 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#0	SAMPLE#1	101.47	%		80.000		143/44- JAN 2002	CAPPUCIO	MACCARI
1	SAMPLE#2	102.53	%		80.000		143/44- JAN 2002	CAPPUCIO	MACCARI
	SAMPLE#3	104.56	%		80.000		143/44- JAN 2002	CAPPUCIO	MACCARI
	SAMPLE#4	106.96	%		80.000		143/44- JAN 2002	CAPPUCIO	MACCARI
	SAMPLE#5	101.34	%		80.000		143/44- JAN 2002	CAPPUCIO	MACCARI
	SAMPLE#6	98.51	%		80.000		143/44- JAN 2002	CAPPUCIO	MACCARI
	DISSOLUTION	102.63	%		80.000		143/44- JAN 2002	CAPPUCIO	MACCARI
	STANDARD DEVIATION	2.917		std:1			143/44- JAN 2002	CAPPUCIO	MACCARI
	C.V.	2.84	%				143/44- JAN 2002	CAPPUCIO	MACCARI
	MIN. DISSOLUTION VALUE	98.51	%		80.000		143/44- JAN 2002	CAPPUCIO	MACCARI
	MAX. DISSOLUTION VALUE	106.96	%		80.000		143/44- JAN 2002	CAPPUCIO	MACCARI

# PHARMACIA

Laboratory  
Information  
Management  
System

## Integrated Analytical Bulletin

### Pharmaceutical Development

Submission Id: 115473

Sample ID (Request No.): 20020167

User sample ID (lot): 1B3G03

Sample Date : 18/01/2002

Sample Type: SU010398 (PNUJ290940AD) CAPSULE

25/MG

Stability Study Start Date : 18/10/2001

Stability Protocol Number : 200-1017 - Manufacturer : PDI/Pharmacia Nerviano

Samples Notes:

Observations on the Method 083GCO024#0, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS

THAN 0.05%

Total correlated substances: 0.41%

RRT

Area (%)

0.94	0.05
1.00	99.60 (SU 11248)
1.97	0.07
2.06	0.29

Approved by: MACCARI  
Approval date: 04-feb-2002

svbollainTEGRATED.rdf

Printed 20-MAR-2002 05:47 PM

Page 3 / 3

by MARIANI



**Pharmacia & Upjohn**  
Pharmaceutical Development

**Integrated Analytical Bulletin**

Laboratory  
Information  
Management  
System

Bulletin ID: 115229

Sample ID (Request No.): 20013300

User sample ID (lot): 183C03

Sample Date : 10/11/2001

Sample Type: STANDARD (PNU21004) CAPSULE

25MG

Stability Study Start Date : 19/10/2001

Stability Protocol Number: 2014017 - Manufacture P05/Pharmacia Nettuno

Samples Notes:

Batch Manufacture Date : 17/09/2001

Stability Study: 183C03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/80% RH.

Tint Label: 1 MONTH

Preparation purpose: CLINICAL SUPPLY + STABILITY

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
08350020	CAPSULE APPEARANCE	CONFORMS	bt				14320 NOV 2001	CAPPUCIO	MACCARI
0	CAPSULE COLOR	CONFORMS	bt				14320 NOV 2001	CAPPUCIO	MACCARI
	CONTENT APPEARANCE	CONFORMS	bt				14320 NOV 2001	CAPPUCIO	MACCARI
	CONTENT COLOR	CONFORMS	bt				14320 NOV 2001	CAPPUCIO	MACCARI
	SMELL	CONFORMS	bt				14320 NOV 2001	CAPPUCIO	MACCARI
083500201	(HPLC TITER) SAMPLE#1	98.783	%		90.000	- 110.000	14320 NOV 2001	CAPPUCIO	MACCARI
S	(HPLC TITER) SAMPLE#2	96.218	%		90.000	- 110.000	14320 NOV 2001	CAPPUCIO	MACCARI
	(HPLC TITER) SAMPLE#3	95.910	%		90.000	- 110.000	14320 NOV 2001	CAPPUCIO	MACCARI
	AVERAGE TITER (HPLC)	96.3	%		90.000	- 110.000	14320 NOV 2001	CAPPUCIO	MACCARI
083500200	TOTAL CORRELATED SUBSTANCES	0.306	%				14320 NOV 2001	CAPPUCIO	MACCARI
0	IMPURITIES NOT LESS THAN 0.05% SEE TEXT		%				14320 NOV 2001	CAPPUCIO	MACCARI
083500200	(HUMIDITY) SAMPLE #1	1.8	%				14320 NOV 2001	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #2	1.6	%				14320 NOV 2001	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #3	1.7	%				14320 NOV 2001	CAPPUCIO	MACCARI
	AVERAGE HUMIDITY	1.7	%				14320 NOV 2001	CAPPUCIO	MACCARI



**Pharmacia & Upjohn**  
Pharmaceutical Development

**Integrated Analytical Bulletin**

Laboratory  
Information  
Management  
System

Batch Number: 115029

Sample ID (Request No.): 20013300

User sample ID (lot): K53013

Sample Date: 18/11/2001

Sample Type: SOLUSION (PVA/25%URAD) CAPSULE

25MG

Stability Study Start Date: 18/11/2001

Stability Protocol Number: 2001017 - Manufacturer: P&G/Hannover/Mengen

Samples Notes:

Batch Manufacture Date: 17/06/2001  
Stability Study: 035633  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Condition: 25°C/80% R.H.  
The Label: 1 MONTH  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:

**APPROVED**

Status:

**COMPLETE**

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analysis	Analyst	Verifier
0356330140 (SAMPLE #1		95.2	%	%	90.000	14320 NOV 2001	CAPPUCIO	MACCARI	
1 (SAMPLE #2		81.8	%	%	80.000	14320 NOV 2001	CAPPUCIO	MACCARI	
(SAMPLE #3		92.4	%	%	90.000	14320 NOV 2001	CAPPUCIO	MACCARI	
(SAMPLE #4		48.0	%	%	80.000	14320 NOV 2001	CAPPUCIO	MACCARI	
(SAMPLE #5		89.0	%	%	80.000	14320 NOV 2001	CAPPUCIO	MACCARI	
(SAMPLE #6		94.7	%	%	90.000	14320 NOV 2001	CAPPUCIO	MACCARI	
DISSOLUTION		90.35	%	%	80.000	14320 NOV 2001	CAPPUCIO	MACCARI	
STANDARD DEVIATION		5.25		#d-1			CAPPUCIO	MACCARI	
C.V.		5.81	%	%			CAPPUCIO	MACCARI	
MINIMUM DISSOLUTION VALUE		81.8	%	%	80.000	14320 NOV 2001	CAPPUCIO	MACCARI	
MAXIMUM DISSOLUTION VALUE		96.24	%	%	90.000	14320 NOV 2001	CAPPUCIO	MACCARI	



**Pharmacia & Upjohn**  
Pharmaceutical Development

**Integrated Analytical Bulletin**

Laboratory  
Information  
Management  
System

Submission #: 115029

Sample ID (Request No.): 20013300

User sample ID (lot): #3G03

Sample Date: 1/11/2001

Sample Type: SDU#388 [PMU/2500HAED] CAPSULE

25MG

Stability Study Start Date: 1/14/2001

Stability Protocol Number: 20010117 - Manufacturer: P&G/Pharmacia Novartis

Samples Notes:

Batch Manufacture Date: 7/7/00/2001

Stability Study: 105G03

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/90% U.R.

Time Label: 1 MONTH

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:  
**APPROVED**

Status:  
**COMPLETE**

Observations on the Method 083GC002#00, component: CAPSULE APPEARANCE

Brick red colored capsule containing yellow-orange powder

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.306%

RRT	Area %
0.65	0.090
1.00	09.60 (SD 11245)
2.15	0.976
2.17	0.140

Approved by: **MAGGARI**  
Approval date: 30-JAN-2001



**Pharmacia & Upjohn**  
Pharmaceutical Development

**Integrated Analytical Bulletin**

Laboratory  
Information  
Management  
System

Submission Id: 116031

Sample ID (Request No.): 20010302

User sample ID (lot): **883 G03**

Sample Date : 18/11/2001

Sample Type: 801013888 (PMID3888) CAPSULE

25MG

Stability Study Start Date : 18/11/2001

Stability Protocol Number : 2001017 - Manufacturer : P&G Informatica Nortelano

Samples Notes:

Condition: **APPROVED**  
Status: **COMPLETE**  
Preparation purpose: CLINICAL SUPPLY + STABILITY

Batch Manufacture Date : 17/09/2001  
Stability Study #883 G03  
Pack Type: 30 ml PLASTIC BOTTLE  
Storage Conditions: 40°C/75% RH.  
Time Label: 1 MONTH

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data	Analytes	Analyst	Verifier
00365101001 (HPLC TITER) SAMPLE#1	CONFORMS	97.43	%		90.000	- 110.000	14320 NOV 2001	CAPPUCCIO	MACCARI	
00365101002 (HPLC TITER) SAMPLE#2	CONFORMS	98.08	%		90.000	- 110.000	14320 NOV 2001	CAPPUCCIO	MACCARI	
00365101003 (HPLC TITER) SAMPLE#3	CONFORMS	96.23	%		90.000	- 110.000	14320 NOV 2001	CAPPUCCIO	MACCARI	
AVERAGE TITER (HPLC)		97.25	%		90.000	- 110.000	14320 NOV 2001	CAPPUCCIO	MACCARI	
00365101000 TOTAL CORRELATED SUBSTANCES	0.45	%	%				14320 NOV 2001	CAPPUCCIO	MACCARI	
00365101000 IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	%	%				14320 NOV 2001	CAPPUCCIO	MACCARI	
00365101000 (HUMIDITY) SAMPLE #1	1.8	%	%				14320 NOV 2001	CAPPUCCIO	MACCARI	
(HUMIDITY) SAMPLE #2	1.9	%	%				14320 NOV 2001	CAPPUCCIO	MACCARI	
(HUMIDITY) SAMPLE #3	1.5	%	%				14320 NOV 2001	CAPPUCCIO	MACCARI	
AVERAGE HUMIDITY	1.88	%	%				14320 NOV 2001	CAPPUCCIO	MACCARI	

**Pharmacia & Upjohn**  
Pharmaceutical Development



**Integrated Analytical Bulletin**

Laboratory  
Information  
Management  
System

Submission ID: 115031

Sample ID (Request No.): 200113302

User sample ID (lot): 163003

Sample Date: 16/11/2001

Sample Type: SU010308 (FNU280043AD) CAPSULE

25MG

Stability Study Start Date: 18/10/2001

Stability Protocol Number: 2001017 - Manufacturer: P&G Healthcare Worldwide

Samples Notes:

Batch Manufacture Date: 17/08/2001

Stability Study: 163003

Pack Type: 30 ml PLASTIC BOTTLE

Storage Conditions: 40°C/75% UR.

Time Label: 1 MONTH

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analysis	Analyst	Verifier
0330601#0	SAMPLE #1	94.7	%		80.000		143/20 NOV/2001	CAPPUCIO	MACCARI
	SAMPLE #2	92.3	%		80.000		143/20 NOV/2001	CAPPUCIO	MACCARI
	SAMPLE #3	86.2	%		80.000		143/20 NOV/2001	CAPPUCIO	MACCARI
	SAMPLE #4	87.5	%		80.000		143/20 NOV/2001	CAPPUCIO	MACCARI
	SAMPLE #5	87.3	%		80.000		143/20 NOV/2001	CAPPUCIO	MACCARI
	SAMPLE #6	97.3	%		80.000		143/20 NOV/2001	CAPPUCIO	MACCARI
DISSOLUTION		95.3	%	std4	80.000		143/20 NOV/2001	CAPPUCIO	MACCARI
STANDARD DEVIATION		4.102					143/20 NOV/2001	CAPPUCIO	MACCARI
C.V.		4.29	%				143/20 NOV/2001	CAPPUCIO	MACCARI
MINIMUM DISSOLUTION VALUE		87.5	%		80.000		143/20 NOV/2001	CAPPUCIO	MACCARI
MAXIMUM DISSOLUTION VALUE		98.33	%		80.000		143/20 NOV/2001	CAPPUCIO	MACCARI



**Pharmacia & Upjohn**  
Pharmaceutical Development

**Integrated Analytical Bulletin**

Laboratory  
Information  
Management  
System

Submission Id: 113031

Sample ID (Request No.): 20013302

User sample ID (lot): **M3G03**

Sample Date: 13/11/2001

Sample Type: SU010505 (PNU-124840) CAPSULE  
250mg

Stability Study Start Date: 16/03/2001

Stability Protocol Number: 2001017 - Manufacture + PDUFA - India N-010

Samples Notes:

Observations on the Method 083GCC02400, component: CAPSULE APPEARANCE

Brick red colored capsule containing yellow-orange powder

Observations on the Method 083GCC01#00, component: IMPURITIES NOT LESS  
THAN 0.05%

Total correlated substances: 0.145%

RT	Area %
0.06	0.06
1.00	90.85 (ISU-11246)
2.17	0.49

Approved by: **MACCARI**  
Approval date: **30-nov-2001**



**Pharmacia & Upjohn**  
Pharmaceutical Development

**Integrated Analytical Bulletin**

Laboratory  
Information  
Management  
System

Sample Id: 114606

Sample ID (Request No.): 2001350

User sample ID (lot): 153G03

Sample Date: 04/17/2001

Sample Type: SUD01350 (PNU29098-0AD) CAPSULE

25MG

Stability Study Start Date: 10/10/2001

Stability Protocol Number: 2001017 - Manufacturer: Pfizer/Nutras

Samples Notes: Opaque brick red colored capsule in containing yellow-orange powder

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analysis	Analyst	Verifier
01360001#01	APPEARANCE	COMPLIANT	OK				14301 OCT 2001	CAPPUCCIO	MACCARTY
01360001#01	HPLC IDENTIFICATION	POSITIVE	OK				14301 OCT 2001	CAPPUCCIO	MACCARTY
01360001#01	(HPLC TITER) SAMPLE#1	97.31	%		95.000	- 105.000	14301 OCT 2001	CAPPUCCIO	MACCARTY
R	(HPLC TITER) SAMPLE#2	90.10	%		95.000	- 105.000	14301 OCT 2001	CAPPUCCIO	MACCARTY
	(HPLC TITER) SAMPLE#3	98.57	%		95.000	- 105.000	14301 OCT 2001	CAPPUCCIO	MACCARTY
	AVERAGE TITER (HPLC)	99.09	%		95.000	- 105.000	14301 OCT 2001	CAPPUCCIO	MACCARTY
01360001#02	TOTAL CORRELATED SUBSTANCES	0.273	%				14301 OCT 2001	CAPPUCCIO	MACCARTY
D	IMPURITIES NOT LESS THAN 0.05% SEE TEXT		%				14301 OCT 2001	CAPPUCCIO	MACCARTY
01360001#02	(HUMIDITY) SAMPLE #1	1.85	%				14301 OCT 2001	CAPPUCCIO	MACCARTY
	(HUMIDITY) SAMPLE #2	1.76	%				14301 OCT 2001	CAPPUCCIO	MACCARTY
	(HUMIDITY) SAMPLE #3	1.90	%				14301 OCT 2001	CAPPUCCIO	MACCARTY
	(HUMIDITY) SAMPLE	1.9	%				14301 OCT 2001	CAPPUCCIO	MACCARTY

# Pharmacia & Upjohn

Pharmaceutical Development



Laboratory  
Information  
Management  
System

## Integrated Analytical Bulletin

Substance Id: 114605  
 Sample ID (Request No.): 200113156  
 User sample ID (lot): H26363

Sample Date: 07/14/2001  
 Sample Type: Strength (NUC2000/AD) CAPSULE  
 250's

Stability Study Start Date: 10/16/2001

Stability Protocol Number: 2001H17 - Manufacturer: Pophamade Research

Samples Notes: Opaque brick red colored capsule in containing yellow-orange powder

Condition:  
**APPROVED**

Status:

**COMPLETE**

Batch Manufacture Date: 17/09/2001

Stability Study: IR3363

Pack Type: BULK

Storage Condition: TIME ZERO

Time Lived: TIME ZERO

Preparation purpose: CLINICAL SUPPLY + STABILITY

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
08SGS0100	SAMPLE #1	85.23	%	%	80.000	90.000	1430th OCT 2001	CAPPUCIO	MACCARI
1.12	SAMPLE #2	92.79	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	SAMPLE #3	98.54	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	SAMPLE #4	93.46	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	SAMPLE #5	81.52	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	SAMPLE #6	84.74	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	SAMPLE #7	88.32	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	SAMPLE #8	95.91	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	SAMPLE #9	90.61	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	SAMPLE #10	105.5	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	SAMPLE #11	96.31	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	SAMPLE #12	88.12	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	DISSOLUTION	92.62	%	%	80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	STANDARD DEVIATION	7.051	s.d.				14301 OCT 2001	CAPPUCIO	MACCARI
	C.V.	7.59	%				14301 OCT 2001	CAPPUCIO	MACCARI
	MINIMUM DISSOLUTION VALUE	81.52	%		80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI
	MAXIMUM DISSOLUTION VALUE	105.5	%		80.000	90.000	14301 OCT 2001	CAPPUCIO	MACCARI

Printed

30-JAN-2002 10:55 AM

by MACCARI

Page

2 / 6



**Pharmacia & Upjohn**  
Pharmaceutical Development

**Integrated Analytical Bulletin**

Laboratory  
Information  
Management  
System

**Submission Ref:** 114908

**Sample ID (Request No.):** 20010153

**User sample ID (lot):** 43563

**Sample Date:** 01/12/2001

**Sample Type:** SU010593 (NULUS010593) CAPSULE

250 MG

**Stability Study Start Date:** 18/10/2001

**Stability Protocol Number:** 20010117 Manufacturer: PDP-Pharmacia Nederland

**Samples Notes:** Opaque brick red colored capsule containing yellow-orange powder

**Condition:**  
**APPROVED**

**Status:**  
**COMPLETE**

**Batch Manufacture Date:** 17/09/2001

**Storage Sample:** 151093

**Pack Type:** BULK

**Storage Condition:** TIME ZERO

**Time Label:** TIME ZERO

**Preparation purpose:** CLINICAL SUPPLY + STABILITY

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analysis	Analyst	Verifier
1	TOTAL AEROBIC BACTERIA	IN LIMITS	1x1		14301 OCT 2001		CAPPUCCIO	MACCARI	
	ESCHERICHIA COLI	ABSENT	1x1		14301 OCT 2001		CAPPUCCIO	MACCARI	
	SALMONELLA	ABSENT	1x1		14301 OCT 2001		CAPPUCCIO	MACCARI	
	FUNGI	IN LIMITS	1x1		14301 OCT 2001		CAPPUCCIO	MACCARI	
	STAPHYLOCOCCUS AUREUS	ABSENT	1x1		14301 OCT 2001		CAPPUCCIO	MACCARI	
	ENTEROBACTERIA	IN LIMITS	1x1		14301 OCT 2001		CAPPUCCIO	MACCARI	



**Pharmacia & Upjohn**  
Pharmaceutical Development

**Integrated Analytical Bulletin**

Laboratory  
Information  
Management  
System

Submission Id: 114006  
Sample ID (Request No.): 20013158  
User Sample ID (lot): 1015G03

Sample Date : 01/11/2001  
Sample Type: SLURRIES (PR1233942D) CAPSULE  
25MG

Stability Protocol Number : 20010717 - Manufacturer : PDRPharmacia Novartis

Samples Notes: Opaque brick red colored capsule in containing yellow-orange powder

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analysis	Analyst	Verifier
1	CONTENT UNIFORMITY#1	\$8.598	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY#2	\$8.290	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY#3	\$8.526	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY#4	\$7.364	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY#5	100.192	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY#6	100.479	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY#7	101.103	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY#8	\$8.637	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY#9	\$8.917	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY#10	100.852	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY	\$9.54	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	STANDARD DEVIATION	1.192	s.d.-1				14301 OCT 2001	CAPPUCCIO	MACCARI
	C.V.	1.2	%				14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY MAX	101.183	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI
	CONTENT UNIFORMITY MIN	\$7.364	%		85.000	- 115.000	14301 OCT 2001	CAPPUCCIO	MACCARI



**Pharmacia & Upjohn**  
Pharmaceutical Development

## Integrated Analytical Bulletin

Laboratory  
Information  
Management  
System

Submission ID: 114906  
Sample ID (Request No.): 20013158

User sample ID (lot): **083GUS3**

Sample date: 01/14/2001

Sample Type: **SOLID (PNU200001D) CAPSULE**

Stability Study Start Date: 18/01/2001

Stability Protocol Number: 2001057 - Manufacturer: PNUPharmaceuticals, New Jersey

Samples Notes: Opaque brick red colored capsule containing yellow-orange powder

Observations on the Method 083GDS0#\_12, component: DISSOLUTION

Dissolution after 15 minutes equal to: 66% (min. 33%-max. 88.7%)

Dissolution after 30 minutes equal to: 92.6% (min. 31.5%-max. 105.5%)

Dissolution after 45 minutes equal to: 97.5% (min. 88%-max. 101.9%)

Observations on the Method 083GMC01#01, component: TOTAL AEROBIC BACTERIA

Total bacterial count < 20 Ufc/g

Observations on the Method 083GMC01#01, component: ENTEROBACTERIA

Enterobacteria < 10 Ufc/g

Observations on the Method 083GMC01#01, component: FUNGI

Fungi (Yeast + Molds) < 10 Ufc/g

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.273%

RT	Area %
0.58	0.054
0.61	0.052
1.00	98.727 (SU 11240)
1.08	0.107
2.19	0.050

Observations on the Method 083GTC16#01\_IR, component: (HPLC TITER)  
SAMPLE #3

Condition: **APPROVED**  
Status: **COMPLETE**

Batch Manufacture Date: 7/19/2001  
Stability Study: 083GUS3

Pack Type: BULK

Storage Condition: TIME ZERO

Time Label: TIME ZERO

Preparation purpose: CLINICAL SUPPLY + STABILITY

5 / 6

Page

Printed 30-JAN-2002 10:55 AM by MACCARI



**Pharmacia & Upjohn**  
Pharmaceutical Development

**Integrated Analytical Bulletin**

Laboratory  
Information  
Management  
System

Submission ID: 144805  
Sample ID (Request No.): 20011368

User sample ID (lot): #3C03

Sample Date: 01/11/2001

Sample Type: SU10035 (PMN12000QUAD) CAPSULE

250MG

Stability Study Start Date: 18/01/2001

Stability Protocol Number: 20010117 • Manufacturer: PDXPharmacie Mexicana

Samples Notes: Opaque brick red colored capsule containing yellow-orange powder

Observations on the Method 083GTC18#01\_IR, component: [HPLC TITER] SAMPLE#3

Observations on the Method 083GTC18#01\_IR, component: AVERAGE TITER (HPLC)

Average titer determined based on nine different samples

Condition: APPROVED  
Status: COMPLETE

Batch Manufacture Date: 17/09/2001

Stability Study: R3C038

Pack Type: BULK

Storage Condition: TIME ZERO

Time Label: TIME ZERO

Preparation purpose: CLINICAL SUPPLY + STABILITY

Approved by: MACCARI  
Approval date: 29-Nov-2001